**ENSR**
2 Technology Park Drive, Westford, Massachusetts, 01886-3140
T 978.589.3000 F 978.589.3100 www.ensr.aecom.com

# Memorandum

Date: November 19, 2007

To: Tim Drexler/USEPA

From: Lisa JN Bradley

Subject: Responses to GeoHydro Comments Dated 9-21-07 re: "Analytical Methodology" and "Data Quality Judgments"

Distribution: Pines Respondents

Responses to Geo-Hydro comments dated September 21, 2007 concerning Larry Jensen's Radiological Data Review of ENSR's Draft *Evaluation of Polycyclic Aromatic Hydrocarbon, Polychlorinated/Dibenzodioxin/Polychlorinated Dibenzofuran, and Radionuclide Data from Yard 520* (Draft Yard 520 Evaluation report) are provided below. As requested, the responses address the "Analytical Methodology" and "Data Quality Judgments" section of the Geo-Hydro comments.

**Analytical Methodology**

1. **"The test does not describe analytical methods."**

   The radionuclide analyses were performed according to the laboratory's (GEL) *Standard Operating Procedure (SOP) for the Determination of Gamma Isotopes* (GL-RAD-A-013, Revision 10) based on the Department of Energy (DOE) EML Procedures Manual (DOE EML HASL-300). This GEL SOP was included as Attachment A-2 in Appendix C, Quality Assurance Project Plan (QAPP), of the *Yard 520 Sampling and Analysis Plan*, June 3, 2005, revised September 2, 2005 (Yard 520 QAPP). The above mentioned GEL SOP and GEL's SOP for *Soil Sample Preparation for the Determination of Radionuclides* (GL-RAD-A-021, Revision 10) are attached to this memorandum.

2. **"When samples were received at a lab were they dried or were samples analyzed as they arrived?"**

   According to GEL's SOP for *Soil Sample Preparation for the Determination of Radionuclides* (GL-RAD-A-021, Revision 10) the samples were dried at 103-105°C and homogenized prior to analysis. (Refer to the attached GEL SOP GL-RAD-A-021 Revision 10).

3. **"What method was used for analysis (gamma spectroscopy, radiochemistry, fluoroscopy, something else?"**

   The method of analysis for the Yard 520 soil samples was gamma spectroscopy. (Refer to the attached GEL SOP GL-RAD-A-013 Revision 10).

**ENSR**
2 Technology Park Drive, Westford, Massachusetts, 01886-3140
T 978.589.3000 F 978.589.3100 www.ensr.aecom.com

**4. "What were the detection limits?"**

The project target detection limits are listed on Table A-5 of the Yard 520 QAPP. The sample specific detection limits for results that were nondetect (flagged with a "U" qualifier) are listed in Table 4 of the evaluation report (refer to the Draft Yard 520 Evaluation report). The sample specific detection limits for all results are listed on the GEL Certificate of Analysis for each sample, which were included in the ENSR Data Validation memorandum.

**5. "Were two aliquots taken from the same sample and both measured (for quality control)? Were there samples measured and then the same sample measured (for quality control)?"**

Laboratory duplicates and field duplicates were included as quality control (QC) checks for the radionuclide analyses.

A laboratory duplicate was performed on sample GP011 and consisted of two separate aliquots taken from the same sample container at the laboratory. The results of the laboratory duplicate analysis met the QC acceptance criteria as indicated in the Yard 520 QAPP.

The field duplicate pair consisted of two separate samples collected by ENSR from the same sample location (GP008). The field duplicate pair consisted of sample IDs GP008ICB092305S and GP008ICB092305D. The results of the field duplicate analysis met the QC acceptance criteria requirements as indicated in the Yard 520 QAPP.

**Data Quality Judgments**

**1. "Most data in Table 4 is to three digits. However, some data is only to two digits (e.g., Uranium-234 for GP008) or to one digit (e.g., Actinium-227 for rGP009). My feeling is that all this data was originally expressed with three digits but was inadvertently rounded to less on the spreadsheet. Data in Table 4 should be reported to the digits reported from the lab."**

For the radionuclides data, the results were reported exactly as reported by the laboratory, except all ending zeros after the decimal place were dropped.

For the inorganic uranium data reported in milligrams per kilograms (mg/kg), the results were rounded to two significant figures from the three significant figures reported by the laboratory.

2. **"For background soils, and contaminated soils containing unprocessed natural radionuclides, one fundamental way to judge if the data has been analyzed well is to review the data and see it the concentrations for each of the three natural radiation series, Uranium (U-238), Thorium (Th-232) and Actinium (U-235) are in equilibrium (each radionuclide concentration in the series has the same numerical value.) Data for GP004-GP013 were grouped by series and are shown in attached Table 1. Some data is acceptable (e.g., Th-232 Decay Series for GP005 which varies from 2.56 – 2.63 picocuries per gram (pCi/g)) and some data is of low quality (e.g., U-238 Decay Series for GP009 which varies from 4.77- 6.81 pCi/g)."**

**ENSR**
2 Technology Park Drive, Westford, Massachusetts, 01886-3140
T 978.589.3000 F 978.589.3100 www.ensr.aecom.com

Based on the quality control parameters reviewed during data validation, the data are valid as reported by the laboratory. The results were considered to be accurate since the calibration and laboratory control sample (LCS) results met the QC acceptance criteria. In addition, the results were considered to have acceptable precision since the laboratory and field duplicate results met the QC acceptance criteria. In addition, even though the relative percent differences (RPDs) between the detected results in the field duplicate pair indicated variability, the detected results were less than five times the minimum detectable activity (MDA); therefore, precision was deemed acceptable.

When comparing the results by series:

- U-238 series (includes U-238, U-234, Th-230, Ra-226, Po-210, and Pb-210);
- U-235 series (includes U-235, Pa-231, Ac-227); and
- Th-232 series (includes Th-232, Ra-228, Th-228)

the results are reasonable based on comparing daughter/parent ratios (e.g. U234/U238, Th-228/Th232) and the overall percent relative differences (%RSD) between the numbers in a series.

The results for the Th-232 series are the best when comparing the Th-228/Th232 ratio because these results are from the same method (alpha spec); whereas the other two series results are from a mixture of gamma spec and alpha spec results (thus we would expect more variation in the results). The overall %RSDs for the results in the U-238 series ranged from 7% to 25% and parent/daughter ratios ranged from 0.8 to 1.5. The results for the field duplicate sample GP008D had the greatest variability with a %RSD of 25% and parent/daughter ratios as high as 1.5, but the results for the parent sample GP008 were had a %RSD of 7% and parent/daughter ratios around one. Overall, the variability between the results is not unreasonable since these are environmental soil samples.

**VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE**

# STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES

## (GL-RAD-A-013 REVISION 10)

APPLICABLE TO METHODS:
EPA 600/4-80-032 Method 901.1 (Modified)
DOE EML HASL-300 (Modified)



# TABLE OF CONTENTS

## 1.0 THE DETERMINATION OF GAMMA ISOTOPES

## 2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Gamma Isotopes in water, soil, urine and miscellaneous matrices.

2.2 Water samples are counted in Marinelli beakers. Soil samples are sealed in aluminum cans, which are counted immediately if Ra-226 is not desired. If Ra-226 is desired, the sealed can is set aside to allow secular equilibrium between Rn-222 and Bi-214. Quantification is done by the abundance of the 609 KeV Bi-214 line.

2.3 This method has been modified from the source method EPA 600/4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 901.1, and the Department of Energy (DOE) EML Procedures Manual source method for Gamma PHA in soils and sediments, HASL-300. For all matrices, similar principles of radiochemical concentration and counting are used.

2.4 This method has been modified on the basis of GEL's Performance Based Measurement System (PBMS).

## 3.0 METHOD APPLICABILITY

3.1 Minimum Detectable Activity (MDA): The MDA is based upon sample volume, instrument background, instrument efficiency, count time and other statistical factors, as well as specific isotopic values such as abundance and half-life.

3.2 Method Precision: Typical relative percent difference (RPD) is less that 20%.

3.3 Method Bias (Accuracy): The method accuracy requirement for gamma, measured by running a laboratory control sample (LCS) with each batch, is 25% of the true value.

3.4 Analysts go through a partnered training program with an already certified analyst for gamma spectroscopy. The analyst receives training on reviewing of standard analytical requirement such as RPD, method bias and technical review of gamma spectra. The analyst can then become qualified to perform the analysis by passing an unknown sample analysis and correctly identifying the isotope(s). Technical training records are maintained electronically by the Quality Systems staff.

## 4.0 DEFINITIONS

4.1 Clean Line: An energy line of an isotope with no known energy lines of other isotopes within 2 KeV. (This excludes daughters that use the same line for quantification.)

4.2 Interfered Line: An energy line of an isotope with one or more energy lines of one or more different isotopes within 2 KeV.

4.3 Single and Double Escape Interference Lines: When high energy gamma lines above 1022 KeV have a large emission rate, it is possible to see single and double escape peaks caused by escape of the 511 KeV annihilation photon(s) from the detector.

4.4 Summation Interference: When high gamma emission rates are seen, sample summation can occur. Prominent in geometries close to detection and in low energy range (i.e., 10,000 gps at 88 KeV, 15,000 gps at 210 KeV), a summation interference can be seen at 88+88=176 KeV, 210+210=420 KeV, 210+88=298KeV.

4.5 False Positive: An isotope that has failed one or more of several tests including half-life, abundance, and energy tolerance (± 2 KeV)

4.6 Abundance Test: The test where the software verifies the presence of 75% of the total abundance of a nuclide in the system library is present. The presence of greater than 75% of the total abundance will cause a nuclide to be identified. The abundance criteria may be reduced to less that 75% for nuclides with several lower abundant photons.

4.7 Energy Tolerance: The test where the software checks the energy line in the spectrum to see if it is within the energy tolerance setting. (The standard setting is 2 KeV.) If it is within this setting then the line is associated with that nuclide. The energy line can be associated with more than one nuclide.

4.8 Half-Life Test: The test to determine if the half-life of the isotope is long enough not to have decayed away. The half-life of the sample is the time from sample date to analysis date plus 1/2 the count time. A limit of no more than eight half-life is the standard setting.

4.9 Key Line: The line chosen by the builder of the library to be the prominent line of the isotope. This line is used in the MDA table for purposes of calculating activity, error and MDA. For non-identified isotopes the key line energy is used as the basis of determining the region used to calculate the activity, error, and the MDA. Usually this line is the most abundant line on a line that is relatively free from interference.

4.10 Abundance (Photon Intensity): The value, usually expressed in percent, given to a photon of specific energy which is emitted during the decay of a radionuclide. The abundance represents the probability of emission of a specific energy photon when a radionuclide is decaying (gamma/disintegration).

4.11 Counting Uncertainty: The error of the reported result due to the counting statistics of the instrument used for qualification.

4.12 Back Scatter: The detection of a count that occurs when an event interacts with counting materials, changes direction, and scatters back to the detector.

## 5.0 METHOD VARIATIONS

Modifications to the procedure are limited to GEL's use of additional isotopes for the daily calibration check and the inclusion of a more stringent calibration and resolution periodicity.

## 6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Keep hands free from moving parts of canning device and Gamma shields.

6.2 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

6.3 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

6.4 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for Handling Biological Materials.

6.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

## 7.0 INTERFERENCES

7.1 Some Gamma isotopes emit gamma lines that may overlap with other isotopes. If the energies of the two isotopes are within 2 KeV, the peaks may not be resolvable and will give a positive bias to the result. This problem is minimized by careful review of the peak search.

7.2 Soil samples may vary in density from the standard used for calibration. This may bias the results due to self-absorption of lower energy (<100 K).

## 8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 100 cc aluminum cans with lids for soil and miscellaneous samples
8.1.2 Gelman Sciences PETRI dish for soil, filters and miscellaneous samples
8.1.3 2 L and 500 mL Marinelli beakers for water samples
8.1.4 Air displacement pipettes.
8.1.5 Can annealing tool
8.1.6 Graduated cylinder

8.2 Reagents, Chemicals and Standards

8.2.1 NIST traceable mixed gamma standard in 100cc aluminum can
8.2.2 NIST traceable 2.0 liter mixed gamma standard in 2 L Marinelli beaker
8.2.3 NIST traceable mixed gamma standard in 0.5 L Marinelli
8.2.4 NIST traceable mixed gamma standard in Gelman Sciences PETRI dish
8.2.5 Standard soil blank
8.2.6 NIST traceable mixed gamma standard of 13-47mm glass fiber filter composites in Gelman Sciences PETRI dish.
8.2.7 NIST traceable aqueous Mixed Gamma Standard: Contains Am-241, Co-60, and Cs-137 as a minimum.
8.2.8 NIST traceable mixed gamma standard of 1-47mm glass fiber
8.2.9 NIST traceable mixed gamma standard frontloaded in BG-300 Impregnated Charcoal Sample Cartridge.
8.2.10 Nitric Acid, reagent grade. (16M)
8.2.11 Hydrofluoric acid, 48%.
8.2.12 Hydrochloric acid, reagent grade. (12M)
8.2.13 Boric acid, 5%. Dissolve 50 grams of $H_3BO_3$ per liter of water

8.3 Instrumentation

8.3.1 High purity germanium detector, with associated electronics and data reduction software
8.3.2 Top loader balance

## 9.0 SAMPLE HANDLING AND PRESERVATION

9.1 For soil samples, 500g of sample should be collected, preferably in a plastic container to avoid breakage.

9.2 For water samples, 2 liters of sample should be collected in a plastic container and preserved to pH2 with Nitric acid.

## 10.0 SAMPLE PREPARATION

10.1 Soil sample preparation.

10.1.1 Prepare the sample for gamma counting in accordance with SOP GL-RAD-A-021 “Soil sample preparation for the determination of radionuclides”.

10.1.2 Fill the appropriate container with sample prepared from step 10.1.1 using the following steps as a guideline:

10.1.2.1 If Ra-226 analysis is required, the sample is placed in a 100cc can for in-growth.

**NOTE:** It is recommended that in-growth be allowed 7 days to quantify Ra-226. Longer intervals can be used at the request of the client. However, shorter in-growth periods may decrease the accuracy of the data. If there is insufficient mass of sample to fill the 100cc can, contact the team or group leader.

10.1.2.2 All homogenized samples shall be placed in the 100cc can. Determine the net weight of the sample. If the net weight is less than 55 grams or greater than 190 grams, contact the team or group leader to determine the appropriate counting container. Record sample weight and date on sample container.

10.1.2.3 If there is insufficient sample to fill the 100cc can, place sample in the 10cc petri dish, cap and seal. Record sample weight and date on sample container.

10.1.2.4 If there is insufficient sample to fill the 10cc petri dish, perform the following digestion process:

10.1.2.4.1 Weigh out an appropriate aliquot into a labeled teflon beaker. Record this weight on the sample container.

10.1.2.4.2 Add 10 mL of concentrated nitric acid to each sample.

10.1.2.4.3 Place samples on medium heat (~300 °F) and cover each sample with a teflon lid. Reflux all samples for 30 minutes.

10.1.2.4.4 Remove teflon lids and add 5 mL concentrated hydrochloric acid and 10 mL hydrofluoric acid to each sample. Cover samples and reflux for 120 minutes.

10.1.2.4.5 Remove teflon lids and allow samples to evaporate to dryness.

10.1.2.4.6 Add 5 mL of concentrated nitric acid and evaporate to dryness.

10.1.2.4.7 Repeat Step 10.1.2.4.6.

10.1.2.4.8 Add 5 mL of concentrated nitric acid to the dry samples. Add 1 ml of 5% boric acid. Place the samples back on the hotplate long enough so that the dried sample dissolves into solution.

10.1.2.4.9 Transfer solution to a 500 mL marinelli beaker and dilute to 500 mL. Record the original sample mass and diluted volume on sample container. Record the original sample mass on batch que sheet.

10.2 Water sample preparation

10.2.1 Mix and measure an appropriate volume into a 2 L or 500 mL Marinelli beaker and record the volume on the batch Que Sheet. If applicable, record the standard identification code, volume and expiration date on the batch Que sheet.

10.3 Urine Sample Preparation

10.3.1 Place a 24-hour urine container (or other suitable container) on a balance and tare the balance.

10.3.2 Transfer the entire volume of the sample received to the tared container and record the volume of sample received.

10.3.3 Add 8 M $HNO_3$ acid to the original sample container (typically 25 – 50 mL). Shake in the container and then heat in a microwave for approximately 30 seconds to remove sample residue from the sides of the sample container.

10.3.4 Add the nitric acid rinse to the 24-hour urine container and record the volume of the original sample plus acid.

10.3.5 Cap and shake the 24-hour urine container to homogenize the sample. Transfer an aliquot (typically 500 mL) of this solution to a Marinelli Beaker.

10.3.6 Record the amount of the original sample, excluding the nitric acid added, on the gamma spec que sheet.

Example: 800 mL is received and 50 mL of 8 M $HNO_3$ is added from the rinse of the sample container. 500 mL is transferred to the Marinelli Beaker. The recorded volume on the que sheet should be (500 mL/850 mL) x 800 mL = 470.6 mL.

10.4 Preparation of miscellaneous matrices

10.4.1 Prepare the sample in accordance with SOP GL-RAD-A-026 "Preparation of Special Matrices for the Determination of Radionuclides."

10.4.2 Once the appropriate section of GL-RAD-A-026 has been performed, prepare the sample for gamma counting by referring to section 10.1.2 above.

## 11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 A blank is performed with each batch. DI Water should be used to prepare the blank.

11.2 A duplicate should be run with each sample batch. Refer to the batch pull sheet to determine the designated batch duplicate sample.

11.3 A matrix spike sample is prepared by adding a known volume of standard directly to the designated sample. Refer to the batch pull sheet to determine the designated batch matrix spike sample.

11.4 A laboratory control sample is prepared by adding a known volume of standard directly to a Marinelli beaker with DI water.

## 12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Refer to "Gamma Spectroscopy System Operating Procedure" (GL-RAD-I-001) for calibration periodicity and instructions.

12.2 Refer to "Counting Room Instrument Maintenance and Performance Checks" (GL-RAD-I-010) for instructions concerning instrument maintenance.

## 13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Place the sample on the detector and count the sample an appropriate amount of time in the gamma shield. See "Gamma Spectroscopy System Operating Procedure" (GL-RAD-I-001) for specific instructions on operating the gamma spectrometers.

## 14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to "Gamma Spectroscopy System Operating Procedure" (GL-RAD-I-001) for instructions concerning the Gamma Spectrometer.

14.2 Refer to "Counting Room Instrument Maintenance and Performance Checks" " (GL-RAD-I-001) for instructions concerning instrument maintenance.

## 15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Data Recording

Record the following information on the Gamma Que Sheet: preparation date, analyst's initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date and time.

15.2 The instrument will report sample pCi/unit according to the following equation:

$$\text{Sample pCi/unit} = \frac{A * d}{2.22 * E * V * B * Ct * ABS}$$

Where:

A = net peak area (counts)
ABS = relative absorption factor
B = abundance (gammas/disintegration)
E = counting Efficiency (counts/gamma)
V = sample volume (grams or liters)
Ct = sample count time (minutes)
d = decay factor = $\frac{1}{e^{-\lambda t}}$

15.3 Counting uncertainty is calculated according to the following equation:

$$\text{pCi/unit} = Ac * 1.96\sqrt{\left(\frac{ef - er}{E}\right)^2 + \left(\frac{pk - er}{pk}\right)^2 + \left(\frac{ab - er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (\text{Decay})}$$

Where:

Ac = Activity from 15.2

$$\text{Decay} = \left(\frac{T_{1/2err}}{T_{1/2}}\right)^2 * \left[\frac{\lambda Er}{1 - e^{-\lambda Er}} - \lambda\left(T_s + Er\right) - 1\right]$$

15.4 The method MDA in pCi/g or pCi/L are calculated according to the following equations:

$$\text{MDA (pCi/unit)} = \frac{d * \left(2.71 + 4.66\sqrt{cpm_b * ct}\right)}{2.22 * E * V * B * ct}$$

Where:

A = net peak area (counts)
ABS = relative absorption factor
B = abundance (gammas/disintegration)
E = counting Efficiency (counts/gamma)
V = sample volume (grams or liters)
ct = sample count time (minutes)
d = decay factor = $d = \frac{1}{e^{-\lambda t}}$

15.5 The absorption factor is calculated by the following equations:

$$I_1 = \frac{\ln((SScpm - Scpm)/ECcpm)}{(((SScpm - Scpm)/ECcpm) - 1)}$$

$$I_0 = \frac{\ln((SSTcpm - STcpm)/ECcpm)}{(((SSTcpm - Scpm)/ECcpm) - 1)}$$

$$ABS = \frac{I_1}{I_0}$$

Where:

SScpm = sample plus the source cpm at the region of interest
Scpm = sample cpm at the region of interest
ECcpm = source cpm on the empty can at the region of interest
ln = natural logarithm
SStcpm = standard plus the source cpm at the region of interest
Stcpm = standard cpm at the region of interest

15.6 The VAX operating system will report the following information with each completed sample:

15.6.1 The nuclide identification report

15.6.2 The minimum detectable activity report

15.6.3 The peak search report.

15.7 The following criteria are used to accept a reported gamma isotope from the NID report:

15.7.1 The peak FWHM should be less than 3 KeV.

15.7.2 The activity of a non-target isotope will not be reported unless the result is greater than the minimum detectable activity and the result is greater than the three sigma uncertainty..

15.7.3 The energy tolerance should be between 2 and 3 KeV.

15.7.4 The sensitivity setting should be between 0.1 and 3. The default setting is 3.

15.7.5 Start channel on peak search should be approximately 50 and end channel should be 4096.

15.7.6 The confidence level setting should be 5.

15.7.7 These settings should not be changed without approval from a group leader.

15.8 The following guidelines are used to accept unidentified lines on the peak search after environmental background subtraction:

15.8.1 The line matches the natural fingerprint of the Uranium-238 or Thorium-232 decay chains (i.e. 63, 75, 93, 239, 295, 352, 511, 609, 1120, etc.).

15.8.2 The line matches as a summation peak from two other lines in the spectrum.

15.8.3 The line has a net area of less than 20.

15.8.4 The line matches as a escape peak from an identified nuclide which emits photons greater than 1022 KeV.

## 16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

Refer to "Analyst and Analytical Methods Validation Procedures" (G-RAD-D-003) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes. Matrix spikes are prepared by spiking a portion of the QC sample with Cs-137 (as a minimum).

16.2.2 For water samples only, a matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{REC} = \frac{\text{spike(pCi/g)} - \text{sample(pCi/g)}}{\text{spikedamount(pCi/g)}} * 100$$

or:

$$\%\text{REC} = \frac{\text{spike(pCi/L)} - \text{sample(pCi/L)}}{\text{spikedamount(pCi/L)}} * 100$$

**NOTE**: Performing a matrix spike on a soil sample would result in direct contamination of the sample, therefore, only water samples require an MS.

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be ≤o 20%. The RPD is calculated as follows.

$$RPD = \frac{\text{high sample (pCi/g)} - \text{low sample (pCi/g)}}{\text{Average (pCi/g)}}$$

or:

$$RPD = \frac{\text{high sample (pCi/L)} - \text{low sample (pCi/L)}}{\text{Average (pCi/L)}}$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The LCS should contain Cs-137 as a minimum. Some clients may request a mixed gamma standard. For soils, a mixed gamma expired calibration source may be used as an LCS. For liquids and filters, spike a blank sample with Cs-137 as a minimum.

16.2.5 The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_pCi/g}}{\text{known\_pCi/g}} * 100$$

or:

$$\text{LCS} = \frac{\text{observed\_pCi/L}}{\text{known\_pCi/L}} * 100$$

16.3 Actions required if the Quality Control Requirements Are Not Met

If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

## 17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The data is transmitted from the laboratory personnel to the reporting personnel as outlined in “Data Review and Validation Procedures” (GL-RAD-D-003):

17.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.1.2 Review the raw data to see if there are any hits not on the requested list. If there are, report to the client by adding the information into LIMS.

A true identification or a “hit” is any isotope greater than 10 pCi/L or 5 pCi/g on the identified nuclide list. The error must also be less than 40% of the result and not have interference by another isotope or have a very short half-life.

17.1.3 Check to see that the required detection limit (RDL) is met if required.

17.1.4 Check hits to see if they are true hits and not an interference or a false positive.

Identifications are classified into two categories: false positives (interference), and true identification (hit). The false positives are rejected by checking the abundance test results for the isotope and by checking last results for the half-life. The result is considered interference and rejected by checking to see if there are any clean lines in sample spectrum for the isotope. If none exist, then the identification is rejected. If the key line has a possible interference and secondary lines do not confirm the activity calculation, the identification is rejected. Isotopes that pass these criteria are accepted as true identifications. The above tests and criteria are standard and will be followed unless directed otherwise by contract, specification or instructions.

17.1.5 Complete the batch checklist.

## 18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

## 19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 After analysis, return sample containers to storage as outlined in "Verifying the maintenance of sample integrity" (GL-LB-E-012).

19.2 Radioactive waste is disposed of as outlined in the Laboratory Waste Management Plan (GL-LB-G-001).

## 20.0 REFERENCES

20.1 USEPA. Prescribed Procedures for Measurement of Radioactivity in Drinking Water. Method 901.1, August 1980.

20.2 Canberra Nuclear Genie System Spectroscopy, Applications and Display User's Guide. Vol. I and II, May 1991.

20.3 EML procedures manual. HASL-300-Ed.25, 1982.

**VERIFY THE VALIDITY OF THIS SOP EACH DAY IN USE**

# SOIL SAMPLE PREPARATION
# FOR
# THE DETERMINATION OF RADIONUCLIDES

## (GL-RAD-A-021 REVISION 10)



## TABLE OF CONTENTS

## 1.0 SOIL SAMPLE PREPARATION FOR THE DETERMINATION OF RADIONUCLIDES

## 2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the preparation of soil samples for radionuclide determination.

## 3.0 INTERFERENCES

This procedure involves drying the soil at a temperature between 103 and 105º C. If that temperature would volatize any components for which an analysis has yet to be run, a separate aliquot must be set aside for such analyses.

## 4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Personnel performing this analytical procedure are trained in and follow the safe laboratory practices outlined in the Safety, Health and Chemical Hygiene Plan, GL-LB-N-001.

4.2 Personnel handling radioactive materials are trained in and follow the procedures outlined in GL-RAD-S-004 for Radioactive Material Handling.

4.3 Personnel handling biological materials are trained in and follow the procedures outlined in GL-RAD-S-010 for Handling Biological Materials.

4.4 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult qualified senior personnel such as a Group or Team Leader.

## 5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Metal cans ~ quart and pint size
5.1.2 Steel balls ~1" diameter
5.1.3 Drying oven or segmented muffle furnace
5.1.4 Sieve Screens, 40 mesh
5.1.5 Paper funnels
5.1.6 100cc aluminum cans
5.1.7 Aluminum loaf pans
5.1.8 SPEX steel grinding containers (various sizes)
5.1.9 Assorted tools and labware

5.2 Reagents, Chemicals and Standards

5.2.1 Sand, clean
5.2.2 Deionized Water (DI water)
5.2.3 1M Nitric Acid, add 63.3 mL to 500 mL Deionized water, dilute to 1000 mL with Deionized water.

5.3 Instrumentation

5.3.1 Paint can shaker, heavy duty
5.3.2 Analytical balance
5.3.3 SPEX Model 8515-115 Shatterbox

## 6.0 SAMPLE COLLECTION & PRESERVATION

A representative sample must be collected from a source of soil and should be large enough (50-100 grams) so that adequate aliquots can be taken to obtain the required sensitivity. The container of choice should be plastic over glass to prevent loss due to break ups during handling. No preservation is required for solid samples.

## 7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to the technical manual provided with the paint shaker for information regarding equipment maintenance.

7.2 Refer to Instruction Manual for SPEX Shatterbox operating instructions.

7.3 The analytical balance should be cleaned after use.

## 8.0 OPERATING PROCEDURES

8.1 Sample Preparation Techniques

8.1.1 Label a clean metal container with the laboratory sample number.

8.1.2 Weigh container. Record weight into the computerized soil prep balance log. Do not tare balance.

8.1.3 Transfer a representative aliquot from the field collection container to the labeled container.

**NOTE**: Sample is dried at 103-105°C. If this temperature will volatize any component for an analysis that has yet to be run, a separate aliquot must be set aside for such analysis.

8.1.4 Enter the pre-oven sample weight into the computerized soil prep balance log. This weight represents the wet sample weight and container weight.

8.1.5 Place the container in a drying oven at a temperature between 103 and 105° C until dry. (Normally overnight).

**NOTE**: The time required to obtain a dry sample will vary depending on the type of material, size of sample, oven type and capacity, and other factors. The influence of these factors generally can be established by good judgment, experience with the materials being tested, and the apparatus being used.

8.1.6 Using heat resistant gloves, remove the sample from the oven and allow to cool.

8.1.7 Record weight into the computerized soil prep balance log. This weight represents the dried sample weight and the container weight.

8.1.8 Homogenize the sample. This is normally accomplished by placing a lid on the container and placing the container in the industrial paint shaker. Depending on the matrix of the soil, it may be necessary to add several stainless steel balls inside the container to assist in homogenizing the sample. The length of time the sample remains on the shaker is dependent on the matrix of the sample, and normally ranges from 5 – 30 minutes. For solid samples that are composed of large particles it may be necessary to reduce the particle size before homogenizing.

8.1.9 Remove the metal container from the shaker and allow to settle for several minutes.

8.1.10 Place the container in the sample preparation hood and remove the lid. If stainless steel balls were added to the container, they should now be removed. If the sample is determined to be radioactive the stainless steel balls are discarded.

8.1.10.1 Rinse excess soil from stainless steel (SS) balls, collect waste and dispose of properly.

8.1.10.2 SS balls are then soaked in 1 M $HNO_3$ for 12 to 24 hours.

8.1.10.3 Remove SS balls from 1 M $HNO_3$ bath and rinse with DI water.

8.1.10.4 Place SS balls in loaf pans and dry in the drying oven at 103 to 105° C.

8.1.10.5 Return SS balls to a new storage container.

8.1.11 Determine an appropriate aliquot based on the analysis required. Normally, depending upon the required analysis, the sample will be passed through a 40 mesh sieve screen. For clients that require a smaller particle size, continue homogenizing samples using steps 8.1.14 through 8.1.18.

8.1.12 Discard the unused portion into the appropriate waste container.

8.1.13 Seal the container. The soil sample is now ready for radiochemical analysis.

8.1.14 Take a portion of remaining sample (more than enough to complete the requested analysis) and further homogenize sample to ~200 mesh. This is accomplished by pulverizing sample in the shatterbox.

**NOTE:** The ~200 mesh is determined based on particle size study in shatterbox instruction manual.

8.1.15 Pulverize sample for a minimum of 5 minutes.

**NOTE**: Refer to Shatterbox Instruction Manual for operating procedure.

8.1.16 To prevent cross-contamination the dish and puck must be decontaminated prior to pulverizing the next sample.

8.1.16.1 Decontaminate the dish and puck by filling with approximately 50 grams of sand. Replace the lid.

8.1.16.2 Place the pulverizer in operation for approximately 5 minutes. Empty the sand from the container.

8.1.16.3 Dampen a clean paper towel with DI water and wipe the dish, puck and lid to ensure that all traces of sand are removed.

8.1.16.4 Frisk the container assembly to scan for decontamination.

8.1.16.5 A decontamination blank is periodically analyzed. When a decontamination blank is analyzed, it is pulverized post cleaning.

**NOTE**: To prevent corrosion, keep the entire container assembly dry.

8.1.17 Record all samples and decontamination blanks in the pulverizer logbook.

## 9.0 CALCULATIONS AND DATA REDUCTION METHODS

The electronic balance program provides documentation of all necessary raw data.

## 10.0 QUALITY CONTROL REQUIREMENTS

10.1 Method Specific Quality Control Requirements

10.1.1 See the specific isotope operating procedure for instructions concerning method quality control requirements.

10.2 Actions required if the Quality Control Requirements are not met

10.2.1 If any of the quality criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined in GL-QS-E-004 for Nonconformance Identification Control, Documentation, Reporting, and Dispositioning.

## 11.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All data is maintained as quality records in accordance with GL-QS-E-008 for Quality Records Management and Disposition.

## 12.0 LABORATORY WASTE HANDLING AND DISPOSAL

Laboratory waste is handled and disposed in accordance with the Laboratory Waste Management Plan, GL-LB-G-001.

## 13.0 REFERENCES

13.1 C999-90, “Standard Practice for Soil Sample Preparation for Determination of Radionuclides” 1993 Annual Book of ASTM Standards, 12.01, pp. 477-478.